

Form PTO-1390  
P21932.P01U.S. DEPARTMENT OF COMMERCE  
PATENT AND TRADEMARK OFFICE

ATTORNEY'S DOCKET NUMBER

P21932

TRANSMITTAL LETTER TO THE UNITED STATES  
DESIGNATED/ELECTED OFFICE (DO/EO/US)  
CONCERNING A FILING UNDER 35 U.S.C. 371U.S. APPLICATION NO. (If known, see 37 CFR  
1.5)

10/030133

INTERNATIONAL APPLICATION NO.

PCT/EP00/07076

INTERNATIONAL FILING DATE

24 July 2000

PRIORITY DATE CLAIMED

26 July 1999

TITLE OF INVENTION

X-RAY ANODE AND PROCESS FOR ITS MANUFACTURE

APPLICANT(S) FOR DO/EO/US

Matthias FRYDA, Lothar SCHAEFER, and Thorsten MATTHEE

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information.

1. ☒ This is a FIRST submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a SECOND or SUBSEQUENT submission of items concerning a filing under 35 U.S.C. 371.
3. ☒ This is an express request to promptly begin national examination procedures (35 U.S.C. 371(f)).
4. ☒ The US has been elected by the expiration of 19 months from the priority date (PCT Article 31).
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
  - a. ☒ is attached hereto (required only if not communicated by the International Bureau).
  - b. ☒ has been communicated by the International Bureau.
  - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☐ An English language translation of the International Application as filed (35 U.S.C. 371 (c)(2)).
7. ☐ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
  - a. ☐ are attached hereto (required only if not communicated by the International Bureau).
  - b. ☐ have been communicated by the International Bureau.
  - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
  - d. ☐ have not been made and will not be made.
8. ☐ An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ☒ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).  
"Executed"
10. ☐ An English language translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (U.S.C. 371(c)(5)).

**Items 11 to 16 below concern other document(s) or information included:**

11. ☐ Assignee: FRAUNHOFER-GESELLSCHAFT ZUR FÖRDERUNG DER ANGEWANDTEN FORSCHUNG e.V. of München, GERMANY
12. ☐ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
13. ☒ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
14. ☐ A FIRST preliminary amendment.  
☐ A SECOND or SUBSEQUENT preliminary amendment.
15. ☐ A substitute specification.
16. ☐ A change of power of attorney and/or address letter.
17. ☐ Figure of Drawing to be published \_\_\_\_\_
18. ☒ Other items or information:
  - PCT/RO/101-PCT Request(in German).
  - PCT/IPEA/401(in German).
  - PCT/IPEA/408(in German).
  - PCT/IPEA/416(in German).
  - PCT/IPEA/409-with three sheets of amended pages(in German).
  - International Application as published Cover Sheet(in German).
  - PCT/IB/301.
  - PCT/IB/304.
  - PCT/IB/308.
  - PCT/ISA/220(in German).
  - PCT/ISA/210(in German).
  - Claim of Priority.

U.S. APPLICATION NO (If known, see 37 CFR 1.5)

10/030133

INTERNATIONAL APPLICATION NO.

PCT/EP00/07076

ATTORNEY'S DOCKET NUMBER

P21932

19. ☒ The following fees are submitted:

CALCULATIONS

PTO USE ONLY

Basic National Fee (37 CFR 1.492(a)(1)-(5)):

Search report has been prepared by the EPO or JPO. .... \$ 890.00

International preliminary examination fee paid to USPTO (37 CFR 1.482). .... \$ 710.00

No international preliminary examination fee paid to USPTO (37 CFR 1.482) but international search fee paid to USPTO (37 CFR 1.445(a)(2)). .... \$ 740.00

Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO. .... \$1,040.00

International preliminary examination fee paid to USPTO (37 CFR 1.482) and all claims satisfied provisions of PCT Article 33(2)-(4). .... \$ 100.00

ENTER APPROPRIATE BASIC FEE AMOUNT =

\$890.00

Surcharge of \$130.00 for furnishing the oath or declaration later than \_\_\_ 20 \_\_\_ 30 months from the earliest claimed priority date (37 CFR 1.492(e)).

\$

Claims

Number Filed

Number Extra

RATE

Total Claims

- 20 =

X \$18.00

\$

Independent Claims

- 3 =

X \$84.00

\$

Multiple dependent claim(s) (if applicable)

+ \$280.00

\$

TOTAL OF ABOVE CALCULATIONS =

\$890.00

Applicant claims small entity status. See 37 CFR 1.27. The fees indicated above are reduced by 1/2.

\$

SUBTOTAL =

\$890.00

Processing fee of \$130.00 for furnishing the English translation later than \_\_\_ 20 \_\_\_ 30 months from the earliest claimed priority date (37 CFR 1.492(f)).

+

Extension of Time fee in the amount of \$

TOTAL NATIONAL FEE =

\$890.00

Fee for recording the enclosed assignment (37 CFR 1.21(h). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 per property

+

\$40.00

TOTAL FEES ENCLOSED =

\$930.00

Amount to be refunded

\$

Charged

\$

a. ☒ A check in the amount of \$930.00 to cover the above fees is enclosed.b. ☐ Please charge my Deposit Account No. \_\_\_\_\_ in the amount of \$ \_\_\_\_\_ to cover the above fees.c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 19-0089.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO CUSTOMER NO. 7055

AT THE PRESENT ADDRESS OF:

Neil F. Greenblum  
GREENBLUM & BERNSTEIN, P.L.C.  
1941 Roland Clarke Place  
Reston, VA 20191  
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07055

PATENT TRADEMARK OFFICE

SIGNATURE  
Neil F. Greenblum  
NAME

28,394

REGISTRATION NUMBER

P21932.A01

## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicants : Matthias FRYDA et al. )  
 ) Applications Branch  
 Appln. No. : 10/030,133 )  
 )  
 Filed : January 25, 2002 )  
 )  
 For : X-RAY ANODE AND PROCESS FOR ITS MANUFACTURE

**PRELIMINARY AMENDMENT AND COVER LETTER  
 SUBMITTING AMENDED PAGES OF APPLICATION**

Commissioner of Patents and Trademarks  
 Washington, D.C. 20231

Sir:

A copy of the International Preliminary Examination Report - Form PCT/IPEA/409 (hereinafter "Report"), was submitted to the Examiner on January 25, 2002. Applicants note that this Report was drawn on pages of description 1 - 7 and 1 sheet of drawings as originally filed, replacement claims 2 - 16 as filed September 5, 2001, and replacement claim 1 as filed November 13, 2001, and includes as an Annex replacement claims 1 - 16 (in German) submitted September 5, 2001 and replacement claim 1 (in German) submitted November 13, 2001. For the Examiner's convenience, an English translation of the Annex is submitted herewith, in which each sheet is identified on the bottom of the page as "MODIFIED SHEET" along with the submission date of the specific sheet.

10030433-031502

Based upon the submission of the modified sheets of claims, Applicants respectfully request examination on the merits of the application containing pages of description 1 - 7 and 1 sheet of drawings as originally submitted and replacement claims 2 - 16 submitted September 5, 2001 and replacement claim 1 submitted November 13, 2001 (in place of originally filed claims 1 - 22).

Additionally, prior to the examination of the above-identified application including replacement claims 1 - 16, amendment of specification and claims as follows:

**IN THE SPECIFICATION**

*Please amend the paragraphs as follows (Marked-up copies of the amended paragraphs are attached as an Appendix):*

*Please replace paragraph [0002] with the following amended paragraph:*

[0002] In x-ray production, metallic anode material is usually irradiated with electrons. The radiation caused by characteristic electronic transitions exits the apparatus through a window transparent for x-rays. In order to avoid absorption, X-ray production results here at low gas pressures. The transparent window serves to separate the low pressure area from the outside area.

*Please replace paragraph [0010] with the following amended paragraph:*

[0010] However, it has been possible to prove with experiments that these disadvantages could be overcompensated by a diamond substrate. Contrary to expectations, it is possible to work with a much smaller focus with an x-ray anode on a diamond window than it is with

an x-ray anode on a beryllium window. The reason for the overcompensation is that diamond is an excellent heat conductor, so the thermal energy produced can be dissipated with particular efficiency through the diamond substrate. The focal spot therefore heats up less and it is possible to decrease the focus diameter. This leads, as desired, to greater radiation densities. Conversely, exchanging a diamond window for the beryllium window with the same beam density and operating life renders possible a thinner anode with lower absorption of x-radiation.

**IN THE CLAIMS**

***Please amend the claims as follows (Marked-up copies of the amended claims are attached as an Appendix):***

4. (Amended) X-ray anode according to claim 1, characterized in that the anode material is a metal, an alloy or several layers of metal.
5. (Amended) X-ray anode according to claim 1, characterized in that the anode material thickness is between 1  $\mu\text{m}$  and 25  $\mu\text{m}$ .
8. (Amended) X-ray anode according to claim 1, characterized in that the anode material completely covers the window.
9. (Amended) X-ray anode according to claim 1, characterized in that the anode material partially covers the window.
10. (Amended) X-ray anode according to claim 1, characterized in that an intermediate layer is provided between the x-ray anode and the diamond window.

11. (Amended) X-ray anode according to claim 1, characterized in that the intermediate layer is an adhesion-promoting layer.

12. (Amended) X-ray anode according to claim 1, characterized in that the intermediate layer is a radiation filter.

13. (Amended) X-ray anode according to claim 1, characterized in that a temperature sensor is provided.

15. (Amended) Use of an x-ray anode according to claim 1 for x-ray microscopes.

16. (Amended) Use of an x-ray anode according to claim 1 for x-ray units.

**REMARKS**

Entry of the foregoing replacement sheets upon which the International Preliminary Examination Report is based and amendment of the specification and claims are respectfully requested. Applicants note that the instant amendments have been made to generally improve the form of the application and remove multiply dependent claims prior to the calculation of fees.

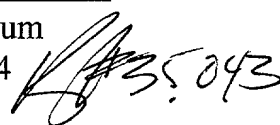
Should there be any questions, the Examiner is invited to contact the undersigned at the below listed number.

Respectfully submitted,  
Matthias FRYDA et al.



Neil F. Greenblum

Reg. No. 28,394



March 19, 2002  
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**APPENDIX**

***Marked-Up Copies of the Amended Paragraphs:***

*Please replace paragraph [0002] with the following amended paragraph:*

[0002] In x-ray production, metallic anode material is usually [impinged on] irradiated with electrons. The radiation caused by characteristic electronic transitions exits the apparatus through a window transparent for x-rays. In order to avoid absorption, X-ray production results here at low gas pressures. The transparent window serves to separate the low pressure area from the outside area.

*Please replace paragraph [0010] with the following amended paragraph:*

[0010] However, it has been possible to prove with experiments that these disadvantages could be overcompensated by a diamond substrate. Contrary to expectations, it is possible to work with a much smaller focus with an x-ray anode on a diamond window than it is with an x-ray anode on a beryllium window. The reason for the overcompensation is that diamond is an excellent heat conductor, so the thermal energy produced can be dissipated with particular efficiency through the diamond substrate. The focal spot therefore heats up less and it is possible to [increase the focus] decrease the focus diameter. This leads, as desired, to greater radiation densities. Conversely, exchanging a diamond window for the beryllium window with the same beam density and operating life renders possible a thinner anode with lower absorption of x-radiation.



***Marked-Up Copies of the Amended Claims:***

4. (Amended) X-ray anode according to [at least one of claims 1 through 3] claim 1, characterized in that the anode material is a metal, an alloy or several layers of metal.

5. (Amended) X-ray anode according to [at least one of claims 1 through 4] claim 1, characterized in that the anode material thickness is between 1  $\mu\text{m}$  and 25  $\mu\text{m}$ .

8. (Amended) X-ray anode according to [at least one of claims 1 through 7] claim 1, characterized in that the anode material completely covers the window.

9. (Amended) X-ray anode according to [at least one of claims 1 through 8] claim 1, characterized in that the anode material partially covers the window.

10. (Amended) X-ray anode according to [at least one of claims 1 through 9] claim 1, characterized in that an intermediate layer is provided between the x-ray anode and the diamond window.

11. (Amended) X-ray anode according to [at least one of claims 1 through 10] claim 1, characterized in that the intermediate layer is an adhesion-promoting layer.

12. (Amended) X-ray anode according to [at least one of claims 1 through 11] claim 1, characterized in that the intermediate layer is a radiation filter.

13. (Amended) X-ray anode according to [at least one of claims 1 through 12] claim 1, characterized in that a temperature sensor is provided.

15. (Amended) Use of an x-ray anode according to [at least one of claims 1 through 14] claim 1 for x-ray microscopes.

16. (Amended) Use of an x-ray anode according to [at least one of claims 1 through 14] claim 1 for x-ray units.

## New Patent Claim 1

1. X-ray anode for microfocus sources in which the anode material is located on a diamond window, characterized in that the thickness of the diamond window is in the range of 300  $\mu\text{m}$  to 2000  $\mu\text{m}$ .

This is followed by the currently valid patent claims 2 through 16.

## New Patent Claims

1. X-ray anode, characterized in that the anode material is located on a diamond window, characterized in that the thickness of the diamond window is in the range of 300  $\mu\text{m}$  to 700  $\mu\text{m}$  or in the range of 700  $\mu\text{m}$  to 2000  $\mu\text{m}$ .
2. X-ray anode according to claim 1, characterized in that it is a polychrystalline diamond window.
3. X-ray anode according to claim 1, characterized in that the diamond window is a monocrystal.
4. X-ray anode according to at least one of claims 1 through 3, characterized in that the anode material is a metal, an alloy or several layers of metal.
5. X-ray anode according to at least one of claims 1 through 4, characterized in that the anode material thickness is between 1  $\mu\text{m}$  and 25  $\mu\text{m}$ .
6. X-ray anode according to claim 5, characterized in that the anode material thickness is between 3  $\mu\text{m}$  and 12  $\mu\text{m}$ .
7. X-ray anode according to claim 6, characterized in that the anode material thickness is 6  $\mu\text{m}$ .
8. X-ray anode according to at least one of claims 1 through 7, characterized in that the anode material completely covers the window.
9. X-ray anode according to at least one of claims 1 through 8, characterized in that the anode material partially covers the window.
10. X-ray anode according to at least one of claims 1 through 9, characterized in that an intermediate layer is provided between the x-ray anode and the diamond window.
11. X-ray anode according to at least one of claims 1 through 10, characterized in that the intermediate layer is an adhesion-promoting layer.
12. X-ray anode according to at least one of claims 1 through 11, characterized in that the intermediate layer is a radiation filter.
13. X-ray anode according to at least one of claims 1 through 12, characterized in that a temperature sensor is provided.
14. X-ray anode according to claim 13, characterized in that the diamond window is provided

as a temperature sensor.

15. Use of an x-ray anode according to at least one of claims 1 through 14 for x-ray microscopes.
16. Use of an x-ray anode according to at least one of claims 1 through 14 for x-ray units.

(12) NACH DEM VERTRAG ÜBER DIE INTERNATIONALE ZUSAMMENARBEIT AUF DEM GEBIET DES  
PATENTWESENS (PCT) VERÖFFENTLICHTE INTERNATIONALE ANMELDUNG

(19) Weltorganisation für geistiges Eigentum  
Internationales Büro



(43) Internationales Veröffentlichungsdatum  
1. Februar 2001 (01.02.2001)

PCT

(10) Internationale Veröffentlichungsnummer  
**WO 01/08195 A1**

(51) Internationale Patentklassifikation<sup>7</sup>: **H01J 35/18**

E.V. [DE/DE]; Leonrodstrasse 54, D-80636 München (DE).

(21) Internationales Aktenzeichen: PCT/EP00/07076

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24. Juli 2000 (24.07.2000)

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(30) Angaben zur Priorität:  
199 34 987.8 26. Juli 1999 (26.07.1999) DE

(71) Anmelder (für alle Bestimmungsstaaten mit Ausnahme von US): **FRAUNHOFER-GESELLSCHAFT ZUR FÖRDERUNG DER ANGEWANDTEN FORSCHUNG**

(72) Erfinder; und

(75) Erfinder/Anmelder (nur für US): **FRYDA, Matthias** [DE/DE]; Eichendorffstrasse 14, D-38110 Braunschweig (DE). **SCHAEFER, Lothar** [DE/DE]; Moorweg 1, D-38527 Meine (DE). **MATTHEE, Thorsten** [DE/DE]; Wiesenweg 11 A, D-38527 Meine (DE).

(81) Bestimmungsstaaten (national): JP, KR, US.

(84) Bestimmungsstaaten (regional): europäisches Patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).

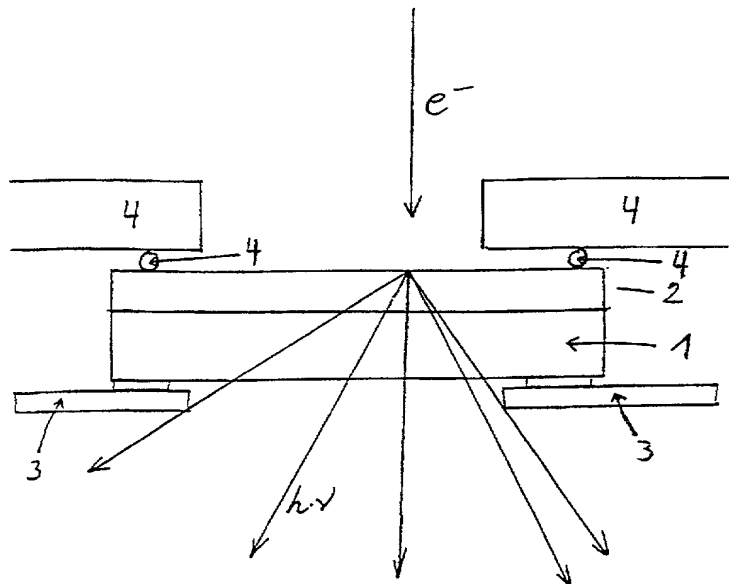
Veröffentlicht:

— Mit internationalem Recherchenbericht.

[Fortsetzung auf der nächsten Seite]

(54) Title: X-RAY ANODE AND METHOD FOR THE PRODUCTION THEREOF

(54) Bezeichnung: RÖNTGENANODE UND VERFAHREN ZU IHRER HERSTELLUNG



(57) Abstract: The invention relates to an X-ray anode and to a method for the production thereof. The inventive X-ray anode is characterized in that the anode material is located as a layer (2) on a diamond window (1). The X-ray anode is preferably used for X-ray apparatuses where an X-radiation is required that is limited as precisely as possible to a defined point in order to achieve a maximum radiation. The inventive X-ray anode is most preferably used in X-ray microscopes in which a high radiation intensity guarantees maximum resolutions.

[Fortsetzung auf der nächsten Seite]

WO 01/08195 A1

Patent Application:  
X-ray anode and Process for its Manufacture

Applicant:  
Fraunhofer-Gesellschaft zur Förderung der angewandten Forschung e.V.

Specification

Technical Field

[0001] The invention relates to an x-ray anode and a process for its manufacture. The x-ray anode according to the invention is preferred for use in x-ray units where the highest possible x-radiation is necessary. It is particularly preferred for use with x-ray microscopes in which a high radiation intensity guarantees the highest resolutions.

Prior Art

[0002] In x-ray production, metallic anode material is usually impinged on with electrons. The radiation caused by characteristic electronic transitions exits the apparatus through a window transparent for x-rays. In order to avoid absorption, X-ray production results here at low gas pressures. The transparent window serves to separate the low pressure area from the outside area.

[0003] Metallic x-ray anodes made of e.g., copper or molybdenum, and a beryllium window in a target angle arrangement are known. There is a certain spacing between the anode and the beryllium window here and they are tilted towards one another. If the x-radiation produced is used for x-ray microscope purposes, this solution has the disadvantage of the resolution being only quite small because of the unavoidable ray divergence between the anode and the object to be imaged. Beryllium is also highly toxic and should therefore be avoided as far as possible as a window material.

[0004] As an alternative to beryllium windows as x-ray exit windows for x-ray units, US 5,173,612 suggests using a diamond window a few 10  $\mu\text{m}$  thick. However, since thicker diamond windows are ruled out because of increased absorption by diamond, these thin diamond windows cause considerable mechanical problems. Thin diamond windows can hardly withstand the pressure differential of approximately  $10^5$  Pa between the low pressure area and the outside area and have to be stabilized by appropriate crosspieces at considerable cost.

**[0005]** Also known are so-called microfocus sources, where the anode material forms a layer on a beryllium window and where the anode is bombarded by an electron beam as strongly focussed as possible. In the case of these microfocus sources, the anode moves closer to the object in optical imaging and the optical resolution can be increased. The more sharply the electron beam bombarding the anode is focussed on the anode, the better the resolution. Disregarding diffractions, a spot focus on the anode would be ideal. However, with a spot focus the problem arises that the energy generated by the electron bombardment causes the material to melt or evaporate, thus reducing its operating life. A thicker anode must be selected to compensate for the evaporation of anode material. However, a thick anode results in the x-radiation being absorbed by the anode material itself. The use of a thicker beryllium window is ruled out for the same reason. Moreover, this solution has the considerable disadvantage that mechanical problems can occur due to the existing pressure differentials, and the microfocus source can easily burst. However, this is particularly harmful in the case of toxic beryllium, where a rupture of the microfocus source leads to undesirable apparatus down-time because of the safety measures for staff protection then required. For these reasons according to prior art spot focussing is possible only to a limited extent.

#### Description of the Invention

**[0006]** The invention is based on the technical problem of producing an x-ray anode that avoids the disadvantages of the prior art as far as possible. The x-ray anode needs to be harmless from a health viewpoint and, in particular, should make it possible to work with a much smaller focus than with the prior art.

**[0007]** The solution of this technical problem is achieved through the features listed in claim 1. The process-related task of producing such an x-ray anode is solved by the features of claim 16. Advantageous embodiments are provided in the dependent claims.

**[0008]** According to the invention it was recognized that the problems could be solved by an x-ray anode where the anode material is on a diamond window.

**[0009]** At first, diamond seems unsuitable as a material for a microfocus source. With an atomic number of  $Z=6$ , diamond absorbs x-radiation more than beryllium at  $Z=4$ . It would therefore be expected that the diamond windows used would have to be thinner than beryllium



windows, entailing the above-mentioned mechanical problems. Moreover, up until now, only beryllium was considered as a window material, since beryllium is a rollable metal from which it is easy to make beryllium windows. According to the prior art, this window serves as a substrate for a metal anode to be applied.

**[0010]** However, it has been possible to prove with experiments that these disadvantages could be overcompensated by a diamond substrate. Contrary to expectations, it is possible to work with a much smaller focus with an x-ray anode on a diamond window than it is with an x-ray anode on a beryllium window. The reason for the overcompensation is that diamond is an excellent heat conductor, so the thermal energy produced can be dissipated with particular efficiency through the diamond substrate. The focal spot therefore heats up less and it is possible to increase the focus. This leads, as desired, to greater radiation densities. Conversely, exchanging a diamond window for the beryllium window with the same beam density and operating life renders possible a thinner anode with lower absorption of x-radiation.

**[0011]** It has been shown that even relatively thick diamond layers can be used advantageously with very thin anodes. In this context, diamond windows are also suitable with thicknesses of between  $50\mu\text{m}$  and  $1000\mu\text{m}$ , or still better between  $300\mu\text{m}$  and  $700\mu\text{m}$ . With such thicknesses, an efficient removal of heat and a good mechanical stability is guaranteed.

**[0012]** According to the present invention, a polycrystalline diamond substrate or diamond window can be used, as well as a monocrystal window. A polycrystalline diamond substrate can be produced particularly simply by means of chemical vapor deposition (CVD), e.g., by hot-filament CVD or microwave CVD. This also makes it possible to produce larger diamond substrates at moderate prices. The deposition of the anode material takes place through a different deposition process, e.g., physical vapor deposition (PVD).

**[0013]** Basically, metals, several layers of metal, or metal alloys can be considered as anode material. The thickness of the anode material should preferably be in the range of between  $1\mu\text{m}$  and  $25\mu\text{m}$ , even better in the range of between  $3\mu\text{m}$  and  $12\mu\text{m}$ , and best of all at  $6\mu\text{m}$ .

**[0014]** The layers do not need to feature constant thicknesses. This means that, e.g., in the case of a disk-shaped microfocus source, the disk thickness does not need to be uniform. The disk can have, e.g., a greater thickness at the edges. The thicknesses given above for the layers should therefore be understood to refer to thicknesses in the focal spot.

**[0015]** In order to ensure that there is always sufficient anode material on the diamond, and that it has not evaporated after a certain number of hours in operation, a temperature sensor can be provided for the x-ray anode according to the invention. A creative possibility here is using the diamond window as a thermistor, i.e., exploiting the temperature dependence of the electrical resistance of the diamond window. After the appropriate calibration, the user has only to set the optimal operating point regarding the desired radiation intensity with a minimal evaporation rate. This makes it easier to avoid thermally-conditioned damage to the x-ray anode according to the invention. Even in the event that part of the anode material has evaporated after a certain number of hours in operation, the diamond window, as an uncommonly thermally stable material, will usually be completely intact. In this case, the remaining anode material can be chemically removed and the diamond window can be recoated in the course of maintenance work. Choosing diamond as a window material thus renders possible a cost-efficient overhaul of the x-ray anode according to the invention, while simultaneously reusing the diamond window.

**[0016]** In its simplest embodiment, the anode material is found holohedrally on the diamond substrate. Depending on the special features of production or of the planned use for the microfocus source, however, it can be sufficient for only part of the diamond layer to be covered by the anode material. Depending on the adhesion of the anode material to the diamond substrate, it can be sufficient to apply the anode material directly on the diamond layer. However, in the case of poor adhesion, an adhesion-promoting intermediate layer can be advantageous. An intermediate layer can likewise be advantageous when as far as possible monochromatic radiation needs to be emitted from the x-ray anode. In this case, the intermediate layer acts as a radiation filter and/or a monochromator.

**[0017]** Tests have further shown that, with the same radiation output, temperature-sensitive samples can be better examined with the x-ray anode according to the invention than with the comparison anode with a beryllium window. Due to the excellent thermal conduction of diamond, the temperatures on the side facing the atmospheric area are lower, which makes it possible to place the samples closer to the window. This in turn results in a better optical resolution.

**[0018]** An exemplary embodiment of the invention is described in greater detail below:

**[0019]** A polycrystalline diamond layer (1) with a thickness of 250  $\mu\text{m}$  is deposited on an

auxiliary substrate using hot-filament CVD. After removing the auxiliary substrate, a tungsten layer (2) with a thickness of 6  $\mu\text{m}$  is deposited on this diamond layer using physical vapor deposition (PVD). The tungsten layer covers the diamond layer completely. The x-ray source is mounted in the housing (4) of a commercial x-ray microscope by means of a clamp (3), with sealing washers (4) being used to ensure a stable vacuum. The only Fig. 1 shows this microfocus source in installed condition. X-radiation  $h\nu$  is produced by localized bombardment of the x-ray anode with electrons  $e^-$ . The maximum achievable radiation density is measured with this x-ray anode. If the diamond layer is replaced with a 500  $\mu\text{m}$  thick beryllium layer under otherwise identical conditions, the radiation density of the x-radiation produced is reduced by a factor of 4. With a diamond layer thickness of likewise 500  $\mu\text{m}$ , the radiation density achievable with the x-ray anode according to the invention would be even better, due to the improved heat dissipation.

2025-03-04 10:00:00

## Patent Claims

1. X-ray anode, characterized in that the anode material is located on a diamond window.
2. X-ray anode according to claim 1, characterized in that it is a polycrystalline diamond window.
3. X-ray anode according to claim 1, characterized in that the diamond window is a monocrystal.
4. X-ray anode according to at least one of claims 1 through 3, characterized in that the thickness of the diamond window is in the range of 50  $\mu\text{m}$  to 2000  $\mu\text{m}$ .
5. X-ray anode according to claim 4, characterized in that the thickness of the diamond window is in the range of 300  $\mu\text{m}$  to 700  $\mu\text{m}$ .
6. X-ray anode according to at least one of claims 1 through 5, characterized in that the anode material is a metal, an alloy or several layers of metal.
7. X-ray anode according to at least one of claims 1 through 6, characterized in that the anode material thickness is between 1  $\mu\text{m}$  and 25  $\mu\text{m}$ .
8. X-ray anode according to claim 7, characterized in that the anode material thickness is between 3  $\mu\text{m}$  and 12  $\mu\text{m}$ .
9. X-ray anode according to claim 8, characterized in that the anode material thickness is 6  $\mu\text{m}$ .
10. X-ray anode according to at least one of claims 1 through 9, characterized in that the anode material completely covers the window.
11. X-ray anode according to at least one of claims 1 through 9, characterized in that the anode material partially covers the window.
12. X-ray anode according to at least one of claims 1 through 11, characterized in that an intermediate layer is provided between the x-ray anode and the diamond window.
13. X-ray anode according to at least one of claims 1 through 12, characterized in that the intermediate layer is an adhesion-promoting layer.
14. X-ray anode according to at least one of claims 1 through 13, characterized in that the intermediate layer is a radiation filter.
15. X-ray anode according to at least one of claims 1 through 14, characterized in that a temperature sensor is provided.

16. X-ray anode according to claim 15, characterized in that the diamond window is provided as a temperature sensor.
17. Process for manufacturing an x-ray anode, particularly for manufacturing an x-ray anode according to one of claims 1 through 16, characterized in that an auxiliary layer is coated with a diamond layer by means of chemical vapor deposition (CVD), and a metallic layer is deposited on this diamond layer.
18. Process according to claim 17, characterized in that the coating of the auxiliary substrate is carried out by means of hot-filament CVD or microwave CVD.
19. Process according to at least one of claims 17 through 18, characterized in that a diamond layer with a thickness of 50  $\mu\text{m}$  to 1000  $\mu\text{m}$  is deposited.
20. Process according to claim 19, characterized in that a diamond layer with a thickness of 300  $\mu\text{m}$  to 700  $\mu\text{m}$  is deposited.
21. Use of an x-ray anode according to at least one of claims 1 through 16 for x-ray units.
22. Use of an x-ray anode according to at least one of claims 1 through 16 for x-ray microscopes.

## Abstract

The invention relates to an x-ray anode and a process for its manufacture. The x-ray anode is characterized in that the anode material is embodied as a layer on a diamond window. The x-ray anode is preferably used with x-ray units which require as selective as possible x-radiation production to achieve as high as possible radiation intensity. Use in x-ray microscopes in which a high radiation intensity guarantees the highest resolutions is particularly preferred.

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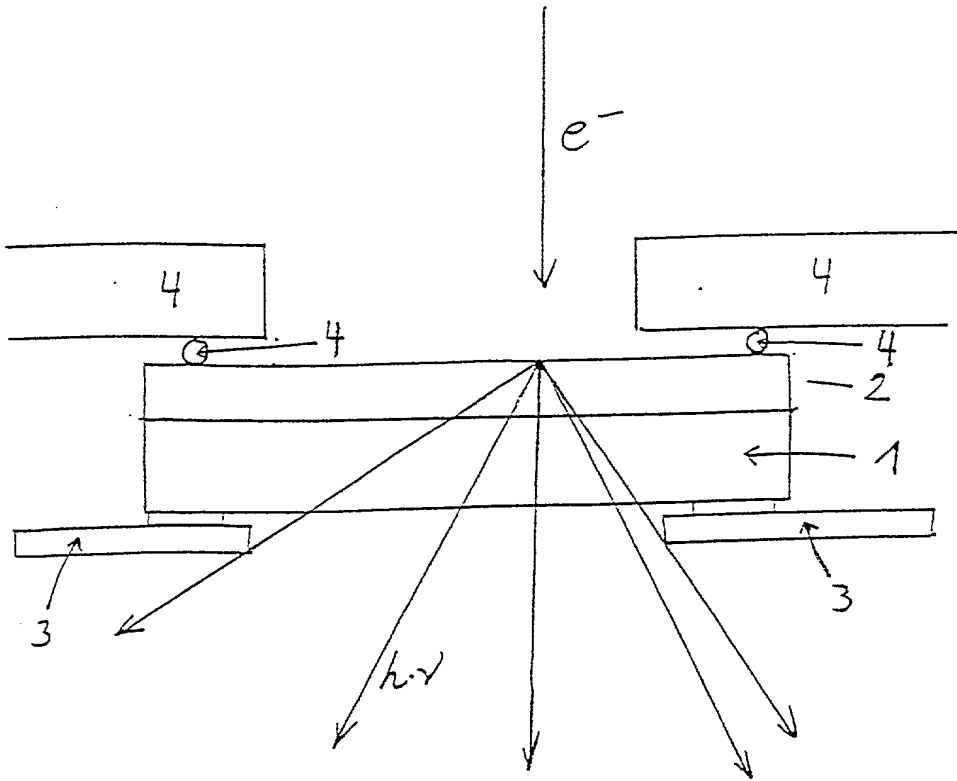


Fig. 1

**Declaration and Power of Attorney For Utility or Design Patent Application**  
**Erklärung für Patentanmeldungen zur Gebrauchseignung und Entwicklung**  
**mit Vollmacht**

**German Language Declaration**

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**RÖNTGENANODE UND VERFAHREN ZU IHRER HERSTELLUNG**

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☒ wurde angemeldet am 24 Juli 2000  
unter der US-Anmeldenummer \_\_\_\_\_  
und wurde am \_\_\_\_\_ abgeändert (falls zutreffend)  
oder

unter der PCT internationalen Anwendungsnummer PCT/EP00/07076  
und wurde am 5 September 2001 and  
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Ich bestätige hiermit, daß ich den Inhalt der oben angegebene Patentanmeldung, einschließlich der Ansprüche, die eventuell durch einen oben erwähnten Zusatzantrag abgeändert wurde, durchgesehen und verstanden habe.

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**Prior Foreign Applications**  
**Frühere ausländische Anmeldungen**

<u>199-34 987 8</u>	<u>Germany</u>
(Number)	(Country)
(Nummer)	(Land)
_____	_____
(Number)	(Country)
(Nummer)	(Land)

<u>26/July/1999</u>
(Day/Month/Year Filed)
(Tag/Monat/Jahr der Anmeldung)
_____
(Day/Month/Year Filed)
(Tag/Monat/Jahr der Anmeldung)

**Priority Claimed**  
**Prioritätsanspruch**

<input checked="" type="checkbox"/> Yes	<input type="checkbox"/> No
Ja	Nein
<input type="checkbox"/>	<input type="checkbox"/>
Yes	No
Ja	Nein

☐ Zusätzliche einstweilige Anwendungsnummern sind im Prioritätsanhang aufgeführt.

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### German Language Utility or Design Patent Application Declaration

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(Aktenzeichen)

(Day/Month/Year Filed)  
(Tag/Monat/Jahr der Anmeldung)

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(Aktenzeichen)

(Day/Month/Year Filed)  
(Tag/Monat/Jahr der Anmeldung)

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(Day/Month/Year Filed)  
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(Status)  
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(Application No.)  
(Aktenzeichen)

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# German Language Utility or Design Patent Application Declaration

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NR. 7077 S. 5/5

## German Language Utility or Design Patent Application Declaration

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Unterschrift des dritten Erfinders

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Sixth Inventor's signature

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NO. 4075

703-716-1180  
CREATION/INVENTION

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